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Fully automated small-scale membrane reactor (MR) system for enzymes and process characterisation

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Introduction

Biocatalysis in membrane reactors offers versatile advantages: Enzymes can be directly used without previous immobilisation, and so the immobilisation procedure, which causes additional costs, development time as well as activity loss can be eliminated, and the advantages of continuous operation can be utilised. However, all commercially available screening and characterisation reactor systems are based on parallel (fed-)batch operated reactors. So, the data collected from such systems can be used only for design of fed-batch applications and has only very limited applicability for designing continuous operations in membrane reactors (MR). The exceedingly important data, such as the estimation of enzyme deactivation phenomena (including enzyme leaching and enzyme adsorption on the membrane, etc.) as well as altering of reaction kinetics in continuous operational mode, fouling phenomena or long-term membrane performance, which are known to be most critical factors for designing EMR-processes (Wöltinger et al., 2005), stay completely obscured. Thus, the aim of this project is the development of a parallel small-scale (<100 mL volume) MR-system, which closes the gap between conventional fed-batch operated lab-scale reactors and fully automated full-scale MR and so to facilitate a reliable early-stage MR processes development.

Problem statement

The developed system has to satisfy versatile requirements: precise monitoring and control of flux (i.e. hydraulic retention time HRT), temperature, pH, catalyst activity and power input must be possible, which at small scale presents a bigger challenge than at industrial scale. All components should be made from inert and sterilisable materials (glass, steel, fluoropolymers), so that no contaminations can occur and all analytic methods can be used. The overall system should be economical.

Results and Discussion

A prototype with preliminarily 2 parallel MRs, based on a modified dead-end membrane test cell (XFUF-047, Millipore Corp.) was designed and constructed. A temperature sensor (1), pH-sensor (2), 4 input PTFE-connectors (3) as well as one permeate outlet (4) were integrated into each MR (Figure 1). A standard size (D = 47 mm) membrane (5) is placed at the bottom of the MR. A stirrer bar (6) is located over membrane surface to prevent membrane fouling.

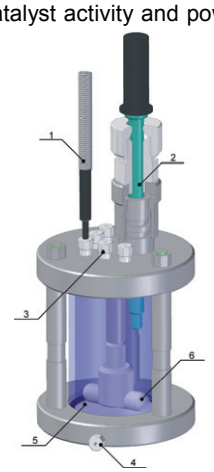

Figure 1: Membrane reactor

Using an integrated PID controller with the feed pressure as the actuator, precise flux control from 3.5-60 L/(m²h) and HRT from 1-18 h with an accuracy of ±1-2 %, respectively, was achieved (Lyagin et al., 2010; 2011). For pH or catalyst activity control, a concept for precise dosing was worked out and realised. It allows liquids (e.g. additional substrates, correction fluids or enzymes) dosing from 0.25-10 mL with ±3.5 % precision (data not shown). For the proof of concept and system's capability, the hydrolysis of N-acetyl-L-methionine (NAM) was studied as

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a model reaction. Figure 2 shows the continuous hydrolysis of NAM with simultaneous enzyme activity control by means of intermittent enzymes dosing with the integrated dosing system.

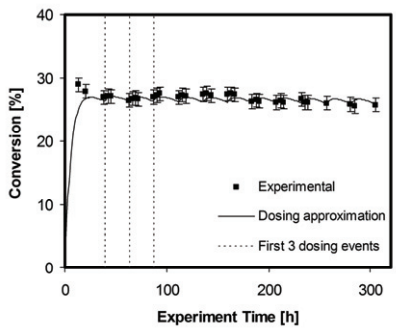

Figure 2: Hydrolysis of NAM during continuous operation with enzyme activity control, HRT=6 h, C_{NAM}=20 mM/L, C_{Enzyme}=3600 U/L, T=30 °C, cellulose membrane with MWCO=10 kDa

The reaction course in figure 2 proves the system's capability: the enzyme activity could be controlled within the target range of ±10 % over the whole duration of the experiment (over 300 h). Thus, the real enzyme consumption per product unit can be directly measured for the tested conditions: T, pH, HRT, enzyme concentration, power input and membrane material. In the next step, the pH control system, based alike on the dosing system, was developed. It is based on a P controller which works in parallel to the PID controller. Figure 3 shows the pH development in the reactor in the first 5 h without pH control and from 5-20 h after the activation of the pH controller.

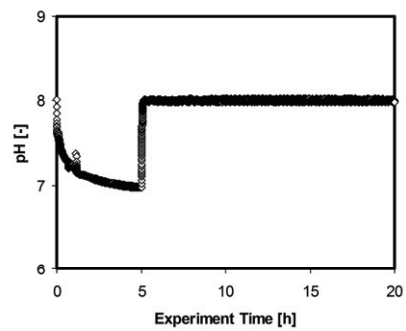

Figure 3: pH-development during continuous operation, process conditions are identical to above

From figure 3 can be seen that the proposed dosing concept could be also implemented for a precise automatic pH control: the target pH of 8.0 was easily maintained within the range of ±0.05 pH units.

Conclusions

The presented system consists of 2 parallel small-scale MR with T, HRT, pH, enzyme activity and power input control. Four PTFE connectors were integrated into the MR, allowing the dosing of additional substrates, enzyme activators etc. Thus, the whole process can be investigated including the measurement of the enzyme consumption per product unit and the membrane long-term performance under tested conditions. Although the system was originally proposed for homogeneous enzymatic catalysis it is also applicable for quasi-homogeneous systems like reverse micelles (Serralheiro et al., 1999) or for polymer-bound catalysts (Laue et al., 2001).

The system is further modified to include new features, more parallel reactors and additional reactions.

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